Monitoring and control of web coating sputtering processes using remote optical spectroscopy gas sensing

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Introduction

The monitoring and control of the vacuum coating process is of upmost importance for consistent, high quality end-product. Monitoring of the gases present within the vacuum chamber is an essential component of this. Every process will at least have one pressure gauge and some will make use of a Residual Gas Analyser (RGA) to monitor the condition of the base vacuum before the start of the process. Very few systems will monitor the gases present during the process itself: this is due to high cost and complexity of differentially pumped RGAs that can operate at process pressures. For web coating systems this is particularly disadvantageous as the often delicate, chemically complex and variable nature of the web substrate can result in inconsistent deposited film quality without knowing the vacuum environment during coating.

The gas sensing technique of Remote Optical Emission Spectroscopy (ROES), whereby a small plasma is generated inside a sensor in order to detect and monitor gases, can be used directly (and therefore cost effectively) to monitor gases at process pressures. Gencoa Limited have developed a novel sensor, called Optix, based on this technique.

This paper presents results from using this sensor to monitor three processes that are both commonplace and emerging in web coating applications: AlOx reactively sputtered deposition process onto PET web, ALD deposition of Aluminium and Niobium compounds and an investigation of the sensors ability to monitor atmospheric processes.

Remote Plasma Emission Monitoring

Plasma emission monitoring has been used for decades to monitor and study the gases present within a vacuum chamber. The light generated by a plasma can be used to identify the emitting species by their wavelength. Remote Plasma Emission Monitoring (RPEM) uses a plasma in the same manner but uses a small plasma generated within the sensor body. This means that it can be used as a stand-alone sensor, without the need for a plasma in the process. Figure 1 describes schematically the operation of a RPEM sensor.

A simple method of generating a small plasma is to use an inverted magnetron. This is a robust design that is easily cleaned, should the electrodes become contaminated. An intense light signal with a low power (< 1W) can also be achieved due to the plasma being concentrated in a small ring around the anode.

The range over which the plasma generation can be stabilised, and hence the operating range of the sensor, can be extended by using a current control loop. This results in an operational range of $1E^{-6}$ mbar up to 0.5 mbar. Below the lower limit the light intensity is too weak to provide adequate signal to noise ratio, whereas the sensor readings become non-linear above the upper limit.

Crucially, this operating range means that the vast majority of industrial vacuum processes can be monitored directly without resorting to differentially pumped sampling systems.

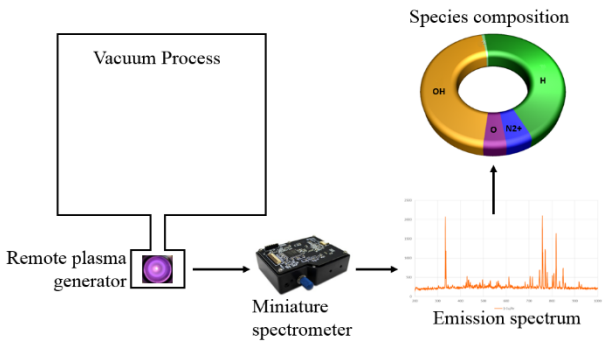

Figure 1 - Schematic of the remote plasma emission monitoring sensing method

Case Study 1 – Atomic layer deposition

Atomic layer deposition (ALD) is an emerging technique for the deposition of thin films. ALD involves the sequential injection of precursors which then form self-limiting layers on the substrate. These precursors are often formed of hydrocarbon groups (such as Trimethylaluminium, TMA) that will quickly contaminate traditional quadrupole RGAs. This, combined with the higher operating pressures of the process, mean that RGAs are rarely used to monitor the process.

RPEM sensing is a promising technique for monitoring ALD processes due to its ability to operate directly at the ALD process pressure and the robustness of its plasma generation. This study investigated the use of the sensing technique for monitoring of various ALD precursors and its robustness during a full deposition cycle lasting over 2 days.

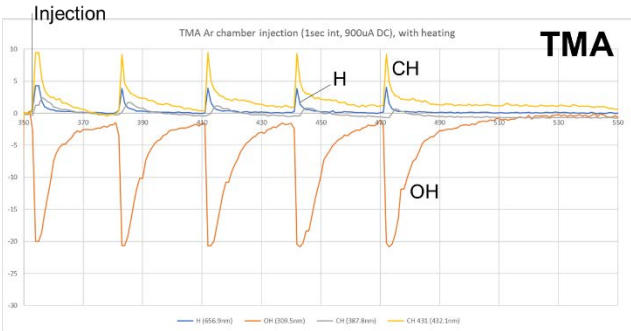

Figure 2 – TMA precursor injection during atomic layer deposition (courtesy of the University of Liverpool)

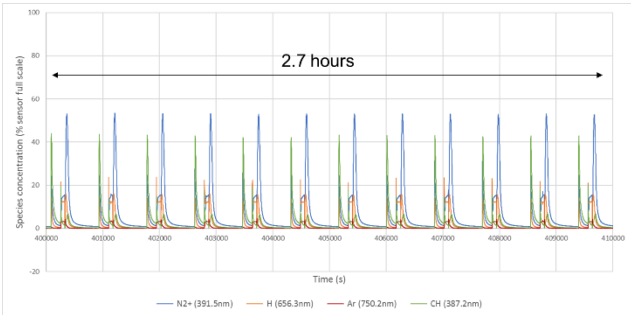

Figure 3 – NbN atomic layer deposition cycle (data courtesy of KEK)

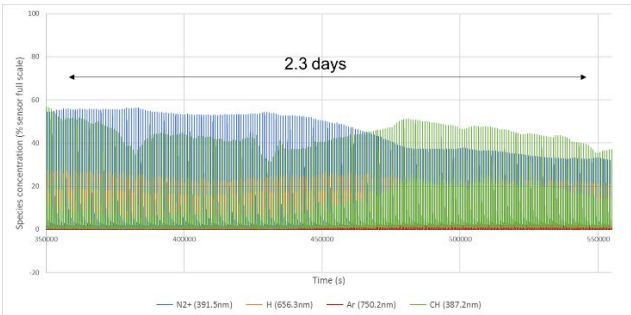

Figure 4 – NbN atomic layer deposition cycle after 2.3 days (data courtesy of KEK).

Figure 2 shows the monitoring of CH, H and OH during injection pulses of TMA. The presence of the TMA can be clearly seen via the increase in H and CH. Furthermore, the gettering effect on water vapour of TMA can be seen through the reduction in OH when the TMA is injected.

Figures 3 and 4 show the results of using the sensor to monitor a NbN deposition process using a plasma enhanced ALD processes. The process uses NH_3 and TrisNb (a hydrocarbon based Nb precursor) to form NbN layers. The sensor remains stable throughout the duration of the deposition cycle, despite the length of over 2 days.

Case Study 2 – Reactive sputter deposition

Reactive sputter deposition uses sputtering of a metallic target material combined with a reactive gas to form thin film compounds. Reactively sputtered AlOx is a commonly deposited thin film used for its barrier layer, dielectric and optical properties. This has many applications for web coating, particularly as a barrier layer. This case study investigates the use of the RPEM sensing technique to characterise an AlOx reactive sputtering process onto PET in terms of the state of the targets, the consumption of reactive gas and the interaction of the process with the surface of the PET substrate.

Figure 5 shows the results of using the sensor to monitor the state of the targets during the initial cleaning phase of the process. When the power is first applied to the target contaminants on its surface are removed via sputtering – this is seen through the increase and then decrease of CO_2, OH and O. Metallic targets will outgas trapped hydrogen continuously during the sputter process, this can also be seen where each subsequent power increase results in a higher level of hydrogen detected.

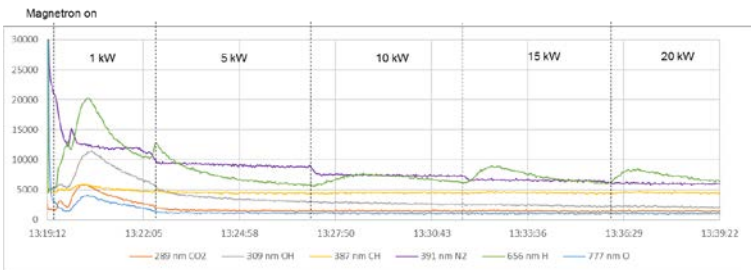

Figure 5 – Monitoring of species produced during Al target cleaning in Ar.

The effect of the sputtering process on the substrate can be seen in Figure 6. When the target poisons (becomes entirely covered with compound) there is a significant increase in un-consumed oxygen. Accompanying this is an increase in CO_2 detected. The CO_2 can be seen to increase an decrease proportionally with the speed of the web, demonstrating that the PET substrate is the source of the CO_2. This is likely due to oxygen ion bombardment of the PET surface – which removes hydrocarbons form the web surface, forming CO_2.

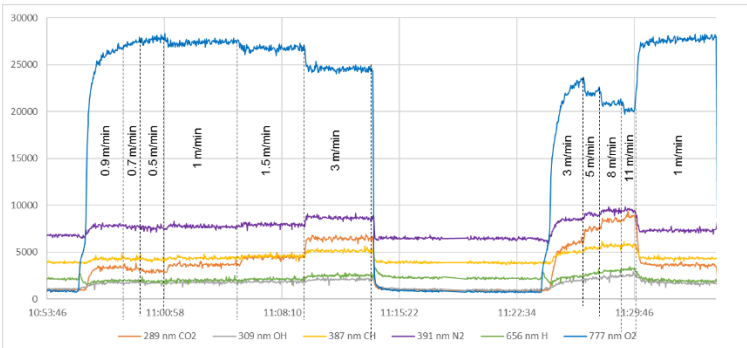

Figure 6 – Monitoring of species during reactive sputtering of AlOx.

Case Study 3 – Atmospheric sampling

The majority of roll-to-roll deposition processes operate below atmospheric pressure, however in some cases atmospheric sampling may be required. In the case of RGAs this requires a pumping system consisting of roughing and turbomolecular pumps and a sampling capillary which is often heated. Such a system is expensive and complex to maintain. A pumping system is also needed for RPEM atmospheric sampling, however due to the requirement to only have the local pressure at the sensor below 0.5 mbar, a much simpler pumping system can be used.

In this case study the objective was to monitor solvents in atmosphere in order to investigate the atmospheric sampling capabilities of the sensor. An experimental setup consisting of an oil sealed rotary pump and a needle valve as the sampling orifice was constructed. A small cap was filled with a solvent and placed near to the orifice. Readings of CH and CO were recorded, this can be seen in Figure 7. It is clear that when the solvent is placed near the orifice readings for CO and CH increase. Furthermore, there are differences in the ratio of CO and CH readings depending on the solvent being sampled. It can be seen that Acetone has a higher proportion of CO measured when compared to Isopropanol due to the presence of the C-O double in the Acetone molecule.

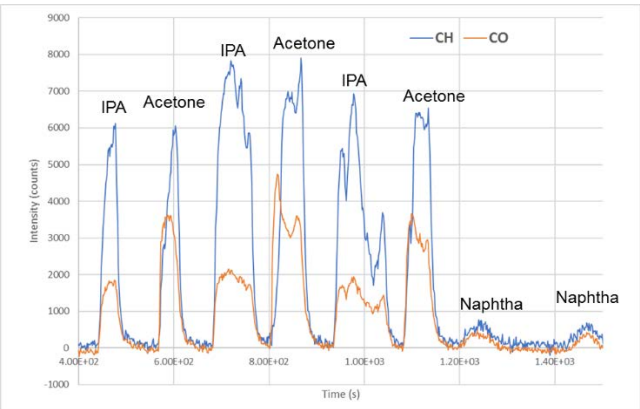

Figure 7 – Atmospheric sampling of solvents, IPA, Acetone and Naphtha.

Conclusions

A sensor that generates a remote plasma to be used for optical spectroscopy can perform monitoring of the relative concentrations of individual species during a vacuum process. This method can be used directly at pressures above $1E^{-4}$ which has some distinct advantages when compared to a differentially pumped RGA sensor. This paper has demonstrated the usefulness of this technique when monitoring potentially contaminating processes such as ALD, as well as commonly used processes such as reactively sputtered AlOx. Furthermore the sensing technique has been shown to be effective for atmospheric sampling when combined with a simple pumping apparatus.

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